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APPLICATION FOR UNITED STATES PATENT

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Invention: PRECIPITATED SILICAS

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SPECIFICATION

PRECIPITATED SILICAS

The present invention relates to precipitated silicas, to the process for the production thereof and to the use thereof in rubber compounds.

BACKGROUND OF THE INVENTION

5　　Precipitated silicas may be incorporated into rubber compounds (S. Wolff, Kautschuk und Gummikunstst. 7 (1988), p. 674). Known silicas may be only very poorly dispersed in rubber compounds, particularly at high filling rates. This poor dispersibility is one of the reasons why elevated sil-

10　ica filling rates are used only rarely in tire compounds. One reason for poor dispersibility may lie in the process by which the precipitated silicas are produced. Drying, poor grinding or also excessively hard granulation may lead to silica particles which cannot readily be dispersed (filler

15　flecks). These flecks may be seen with the naked eye.

Furthermore, silicas are highly polar and therefore have only poor phase compatibility with the non-polar polymers in the rubber compound. This form of dispersion occurs on the basis of the silica aggregates. It may only be

20　evaluated using a light microscope and is known as microdispersion.

Precipitated silicas which may be used as a filler in rubber compounds for tires are known from EP-A 0 520 862.

A precipitated silica is known from published European

25　Patent Application EP-A 0 157 703 which, according to EP-A 0 501 227, may be used as a filler in rubber compounds for tires.

Known precipitated silicas have the disadvantage that they exhibit poor microdispersion.

SUMMARY OF THE INVENTION

30　　The object of the invention is to provide a precipitated silica with optimum phase compatibility with rubber, polymer and good microdispersion.

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The present invention provides a precipitated silica characterized by the following physicochemical properties:

BET surface area	35 to 350 m^2/g
BET/CTAB surface area ratio	0.8 to 1.1
5 Pore volume, PV	1.6 to 3.4 ml/g
Silanol group density (V_2 = NaOH consumption)	6 to 20 ml
Average aggregate size	250 to 1500 nm
CTAB surface area	30 to 350 m^2/g
10 DBP value	150 to 300 ml/100 g
V_2/V_1 by Hg porosimetry	0.19 to 0.46,
preferably	0.20 to 0.23
DBP/CTAB	1.2 to 2.4.

The physicochemical properties are determined using the

15 following measurement methods:

BET surface area	Areameter, Ströhlein, to ISO 5794/ Annex D
Pore volume	Mercury porosimetry to DIN 66 133
20 Silanol group density	in Sears values according to G.W. Sears, Analyt. Chemistry 12, 1982-83 (1956)
Average aggregate size	Photon correlation spectroscopy
25 CTAB surface area	at pH 9 according to Jay, Jan-zen and Kraus in Rubber Chem-istry and Technology 44 (1971), 1287
DBP value	ASTM D 2414-88
30 Hg porosimetry	DIN 66 133

The precipitated silica according to the invention may in particular have the following physicochemical parameters:

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BET surface area (m²/g)	Hg porosimetry (ml/g)	Sears value V_2 NaOH (ml)	Average aggregate size (nm)
35 - 100	2.5 - 3.4	6 - 12	900 - 1500
100 - 150	2.4 - 3.2	8 - 15	400 - 850
150 - 200	1.6 - 2.4	11 - 19	300 - 550
200 - 350	1.6 - 2.3	12 - 20	250 - 520

In a preferred embodiment, the precipitated silica according to the invention has good grindability. This characteristic is represented by the average particle size determined using Malvern laser diffraction (D(4.3)) of ≤ 11 μm, in particular of ≤ 10 μm, measured after grinding on an Alpine Kolloplex pin impact mill (Z 160) at a throughput of 6 kg/h.

The present invention also provides a process for the production of the precipitated silica with the following physicochemical parameters

BET surface area	35 to 350 m²/g
BET/CTAB surface area ratio	0.8 to 1.1
Pore volume, PV	1.6 to 3.4 ml/g
Silanol group density (V2 = NaOH consumption)	6 to 20 ml
Average aggregate size	250 to 1500 nm
CTAB surface area	30 to 350 m²/g
DBP value	150 to 300 ml/100 g
V_2/V_1 by Hg porosimetry	0.19 to 0.46,
preferably	0.20 to 0.23
DBP/CTAB	1.2 to 2.4

which is characterized in that alkali silicate is reacted with mineral acids at temperatures of 60 to 95°C while maintaining a pH of 7.5 to 10.5 and continuously stirring, the reaction is continued to a solids concentration in the precipitation suspension of 90 to 120 g/l, the pH value is adjusted to a value of less than or equal to 5, the
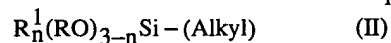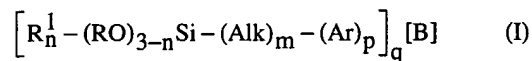
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precipitated silica filtered out, washed, dried and option-
ally ground or granulated.

In a preferred embodiment, customary commercial sodium
water glass may be diluted with water to a pH of 8 to 9 and
5 concentrated sulfuric acid and the same water glass solution
may simultaneously be added to this diluted water glass so-
lution which has an SiO_2 content of 4.9 g/l, while main-
taining the pH value of 8 to 9.

Simultaneous addition of water glass solution and sul-
10 furic acid may be performed over a period (duration of pre-
cipitation) of up to 160 minutes, preferably of greater than
90 minutes, in particular within 30 to 90 minutes.

Depending upon the duration of precipitation, differing
BET surface areas of the precipitated silica may be
15 achieved. Thus, at a duration of precipitation of more than
90 minutes, surface areas of 35 to 150 m^2/g are achieved
and, at a duration of 30 to 90 minutes, surface areas of 150
to 350 m^2/g.

The precipitated silica according to the invention may
20 be modified with organosilanes of the formulas I to III.

$$\left[R_n^1 - (RO)_{3-n} Si - (Alk)_m - (Ar)_p \right]_q [B] \qquad (I)$$
$$R_n^1 (RO)_{3-n} Si - (Alkyl) \qquad (II)$$

or

$$R_n^1 (RO)_{3-n} Si (Alkenyl) \qquad (III)$$

25 in which

B: means -SCN, -SH, -Cl, $-NH_2$ (if q = 1) or
                          $-S_x-$ (if q = 2),

R and R^1:   mean an alkyl group with 1 to 4 carbon
                          atoms, the phenyl residue, wherein all
30   residues R and R^1 may each have the same
                          or different meaning,

R: means a C_1-C_4 alkyl, $-C_1$-C_4 alkoxy group,

n: means 0, 1 or 2,

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Alk:	means a divalent unbranched or branched hydrocarbon residue with 1 to 6 carbon atoms,
m:	means 0 or 1,
5 Ar:	means an arylene residue with 6 to 12 carbon atoms, preferably 6 carbon atoms,
p:	means 0 or 1, providing that p and n do not simultaneously mean 0,
x:	means a number from 2 to 8,
10 Alkyl:	means a monovalent unbranched or branched unsaturated hydrocarbon residue with 1 to 20 carbon atoms, preferably 2 to 8 carbon atoms,
Alkenyl: 15	means a monovalent unbranched or branched unsaturated hydrocarbon residue with 2 to 20 carbon atoms, preferably 2 to 8 carbon atoms.

Modification with organosilanes may be performed in mixtures of 0.5 to 50 parts of organosilanes, related to 100
20 parts of precipitated silica, in particular of 2 to 15 parts, related to 100 parts of precipitated silica, wherein the reaction between the precipitated silica and silane may be performed during compounding (in situ) or outside the compounding process (premodified).

25   In a preferred embodiment of the invention, Bis-(3-[triethoxy-silyl]-propyl)-tetrasulfane may be used as the silane.

The precipitated silica according to the invention may be incorporated into vulcanizable rubber compounds as a re-
30 inforcing filler in quantities of 5 to 200 parts, related to 100 parts of rubber, as a powder, microbeads or granules both with and without silane modification.

One or more of the above-stated silanes may be added,
together with the silica according to the invention, to the
rubber compound, wherein the reaction between the filler and
silane proceeds during the compounding process at elevated
5 temperatures (in situ modification), or in already premodi-
fied form (see, for example, German patent DE 40 04 781),
i.e. the two reactants are reacted outside the actual com-
pounding process.

In addition to compounds exclusively containing the
10 silicas according to the invention with and without or-
ganosilanes according to formulae I to III, the rubber com-
pounds may additionally be reinforced with one or more
fillers having a greater or lesser reinforcing action. It
would primarily be customary in this connection to use a
15 blend of carbon blacks (for example furnace, gas, flame,
acetylene blacks) and the silicas according to the inven-
tion, with or without silane, but also of natural fillers,
such as for example clays, siliceous chalks, other commer-
cial silicas and the silicas according to the invention.
20 The blending ratio, as with the quantity of or-
ganosilane added, depends in this connection on the range of
properties to be achieved in the finished rubber compound.
A ratio of 5-95% between the silicas according to the
invention and the other above-stated fillers, i.e., the
25 silica according to the invention is 5-95% by weight of the
mixture of fillers, is conceivable and is also achieved in
this context.

Apart from the silicas according to the invention, the
organosilanes and other fillers, the elastomers comprise a
30 further important constituent of the rubber blend. The
silicas according to the invention may be used in any types
of rubber which may be vulcanized with accelerators/sulphur
or also peroxide curable rubbers. Rubbers which may be

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cited in this connection are natural or synthetic elastomers, oil-extended or not, as a single polymer or blend with other rubbers such as for example natural rubbers, butadiene rubbers, isoprene rubbers, butadiene/ styrene rubbers, in

5 particular SBR produced using the solution polymerization process, butadiene/acrylonitrile rubbers, butyl rubbers, terpolymers of ethylene, propylene and unconjugated dienes.

The following additional rubbers may moreover also be considered for use in rubber compounds with the stated

10 rubbers:

        carboxyl rubbers, epoxy rubbers, trans-polypentenamers, halogenated butyl rubbers, 2-chlorobutadiene rubbers, ethylene/vinyl acetate copolymers,

15   ethylene/propylene copolymers, optionally together with chemical derivatives of natural rubber and modified natural rubbers.

Customary further constituents such as plasticizers,

20 stabilizers, activators, pigments, antioxidants and processing auxiliaries in customary quantities are also known.

The silicas according to the invention, with and without silane, may be used in any rubber applications, such as

25 for example tires, conveyor belts, seals, V-belts, tubes, shoe soles etc..

Dispersion, i.e. the distribution of a substance (filler) in a polymer compound, is of vital significance to the subsequent properties of the article containing this

30 substance. Tear strength values in particular (tensile strength, elongation at break, tear propagation resistance), but also hysteresis and abrasion values are highly dependent upon dispersion (C.W. Schweitzer, W.M. Hess, J.E. Callun,

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Rubber World, 138, No. 6, 809 (1958) and 139, No. 1, 74 (1958)), (W.M. Hess, F.P. Ford, Rubber Chem. Tech., 36, No. 5, 1191 (1963)).

The significance of this parameter to rubber properties is accompanied by a lack of options for exactly measuring it, or many of the most commonly used methods allow only a subjective examination and assessment of dispersion.

The most widely used methods for measuring dispersion are described in ASTM D 2663-88 and were all developed to measure the dispersion of carbon black in rubber, but may however also be used to measure the dispersion of silica-filled compounds, provided that the compound contains only this filler and not blends, for example carbon black and silica.

One of the three methods described in the above-mentioned standard involves visual examination with the naked eye or slight magnification under the microscope and photographic recording of the vulcanizate samples, wherein the result is assessed against 5 standard photographs using a numbered scale from 1 to 5.

Another method is to count filler agglomerates with a size of ≤ 5 μm. To this end, a microtome section of the vulcanizate is prepared and the percentage of the area occupied by these agglomerates is determined by transmission light microscopy. Here too, dispersion is divided into classes.

The third option described is scanning the surface roughness of the vulcanizate with a needle. In this manner, the number and average height of surface roughness is measured. As with method 2, the roughness factor is converted into a dispersion index from very good to very poor.

The microscopic method (for example 30 times magnification), in which dispersion in the vulcanizate is assessed against a numbered scale from 1 to 10 using 10 standard photographs is presently the most commonly used

5 method, which, although subjective, is rapid, meaningful and most suited to laboratory operation.

Dispersion and its evaluation described above constitute a property of the silica in the polymer compound. In order to be able to predict the subsequent dispersion

10 behavior of a silica in the polymer compound before it is compounded, for example with rubber, the person skilled in the art makes use of the grindability of the silica. In other words, the grindability of the silica and its subsequent dispersion, for example in rubber, largely correlate.

15 This grindability may inter alia be characterized by the energy required to achieve a particular particle fineness, or conversely by the particle fineness achieved when a grinder is operated at the same power and product throughput. The mill used is an Alpine-Kolloplex pin impact mill

20 (Z 160) operated at a constant product throughput of 6 kg/h.

Particle fineness is characterized by the average, volume-weighted particle diameter MTG (D(4.3)) measured by laser diffraction (Malvern Instruments, model 2600c).

Values of ≤ 11 µm, in particular of ≤ 10 µm, are

25 achieved for the silicas according to the invention, which are higher in customary silicas (≥ 12 µm). It has also been found that, due to the process by which the silicas are produced, the silicas according to the invention are already so fine that, in contrast with conventional products, they

30 require no further grinding for many industrial applications and thus also offer economic advantages.

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The present invention also provides vulcanizable rubber compounds which contain the precipitated silicas according to the invention in quantities of 5 to 200 parts, related to 100 parts of rubber. Incorporation of this silica and pro-
5   duction of the compounds containing this silica proceed in the manner customary in the rubber industry in an internal mixer or roll mill. The silica may be presented or used as powder, microbeads or granules. In this respect too, the silicas according to the invention do not differ from known,
10 light silicate fillers.

Due to the above-stated differences, the precipitated silicas according to the invention lead to improved disper- sion properties at a constant surface area compared with standard silicas.

15   The silicas according to the invention also exhibit better properties in relation to some other technically im- portant rubber parameters. The following properties may be mentioned, higher modulus, lower tan δ as a measure of tire rolling resistance, better abrasion resistance, lower T-
20 center values, better wet skid resistance, better rebound resilience, better heat build-up performance and better viscosity.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS
The following examples illustrate the invention:

25 The following substances are used in the examples:

First Latex Crepe	- natural rubber
Ultrasil VN 2	- precipitated silica (Degussa AG) with an N_2 surface area of 125 m^2/g
Ultrasil VN 3	- precipitated silica (Degussa AG) with an N_2 surface area of 175 m^2/g
CBS	- benzothiazyl-2-cyclohexylsulphenamide
TMTM	- tetramethylthiuram monosulphide

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Si 69   - bis(3-triethoxysilylpropyl)- tetra-
sulfane (Degussa AG)

DEG   - diethylene glycol

VSL 1955 S 25 - solution polymerized styrene/ butadi-
ene rubber with a styrene content of 25%
and a vinyl content of 55% (Bayer AG)

DPG   - diphenylguanidine

Vulkanox 4020 - N-(1,3-dimethylbutyl)-N-phenyl-p-
phenylenediamine (Bayer AG)

Protector G 35 - ozone protective wax

ZBED   - zinc dibenzylthiocarbamate

Buna CB 24   - butadiene rubber from Bunawerke Hüls

Naftolene ZD   - aromatic mineral oil plasticizer

Hisil 210   - silica from PPG with an N_2 surface
area of approx. 130 m^2/g

Hisil 255   - silica from PPG with an N_2 surface
area of approx. 170 m^2/g

KS 300   - silica from Akzo with an N_2 surface
area of approx. 125 m^2/g

KS 404   - silica from Akzo with an N_2 surface
area of approx. 175 m^2/g

The following test standards were used:

Test	Unit	Standard
Tensile stress	MPa	DIN 53 504
Compression set B	%	ASTM D 395
Loss angle tan δ	–	DIN 53 513
DIN abrasion	mm^3	DIN 53 516
Firestone ball rebound	%	AD 20 405
Mooney viscosity	ME	DIN 53 523/524
Goodrich flexometer		ASTM D 623 A

Example 1

Production of a silica according to the invention with
an N_2 surface area in the range 100 m^2/g

43.5 m³ of hot water is introduced into a vat and cus-
tomary commercial sodium waterglass (weight modulus 3.42,
density 1.348) is stirred in in a quantity such that a pH of
8.5 is achieved. While maintaining a precipitation

5 temperature of 88°C and pH 8.5, 16,8 m³ of the same water
glass and sulfuric acid (96%) are simultaneously added
within 150 minutes from opposite positions. A solids con-
tent of 100 g/l is produced. Further sulfuric acid is then
added until a pH of < 5 is achieved. The solids are sepa-

10 rated in presses, washed and the pressed paste dried by
spray drying or in a rotary kiln and optionally ground.

The precipitated silica obtained has an N_2 surface area
of 80 m²/g, an aggregate size of 1320 nm and grindability of
10 μm. The Sears value (V_2) is 9.0 ml and Hg porosimetry

15 2.7 ml/g. The CTAB surface area is 75 m²/g. The DBP value
**is 236 ml/100 g. The V_2/V_1 ratio is 0.327. The DBP/CTAB
ratio is 3.5.**

Example 2

Production of a silica according to the invention with an N_2

20 surface area of the 100 - 150 m²/g range

The same procedure is used as in Example 1, with the
exception that a pH value of 9.0 is maintained in the ini-
tial precipitation batch and during precipitation. After
135 minutes, a solids content of 98 g/l is achieved in the

25 precipitation suspension.

The precipitated silica obtained has an N_2 surface area
of 120 m²/g, grindability of 8.8 μm, a Sears value of 9.1 ml
at an aggregate size of 490 nm and an Hg pore volume of
2.85 ml/g. The DBP value is 270 ml/100 g. The CTAB surface

30 **area is 115 m²/g. The V_2/V_1 ratio is 0.27. The DBP/CTAB
ratio is 2.34.**

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Example 3

Production of a silica according to the invention with an N_2 surface area in the 150 - 200 m^2/g range

The same procedure is used as in Example 2, with the

5 difference that the precipitation time is shortened to 76 minutes and the precipitation temperature reduced to 80°C. After this period, a solids content in the precipitation suspension of 100 g/l is achieved. The precipitated silica obtained has the following physicochemical parameters:

10 BET surface area   184 m^2/g

grindability of   8.7 μm

Sears value   15.7 ml

at an aggregate size of 381 nm, Hg pore volume 2.26 ml/g. The CTAB surface area is 165 m^2/g. The DBP value is

15 255 ml/100 g. The V_2/V_1 ratio is 0.2080 to 0.2299. The DBP/CTAB ratio is 1.545.

Example 4

Determination of pore volume by Hg porosimetry on silicas according to the invention compared with some presently

20 known commercial standard silicas

Process: Hg - Porosimetry according to DIN 66 133
injection process 7-500 bar

N_2 surface area (m^2/g): 100 - 150

Product Name	N_2 surface area (m^2/g)	Pore Volume
Hisil 210	130	1.54
KS 300	125	1.98
Ultrasil VN 2	125	1.82
Silica according to the invention (Example 2)	120	2.85

N_2 surface area (m^2/g): 150 - 200

Product name	N_2 surface area (m^2/g)	Pore volume (ml/g)
Hisil 255	170	1.13
KS 404	175	1.66
Ultrasil VN 3	175	1.46
Silica according to the invention (example 3)	184	2.26

The silicas according to the invention have a distinctly higher pore volume.

Example 5

Comparison of Sears value (V_2) as a measure of the OH group density of the silicas according to the invention with standard commercial silicas

N_2 surface area (m2/g): 100 - 150

Product name	N_2 surface area (m^2/g)	V_2 (ml) = NaOH consumption (ml/g)
Hisil 210	130	16.8
KS 300	125	16.1
Ultrasil VN 2	125	15
Silica according to the invention (Example 2)	120	9.1

N_2 surface area (m^2/g): 150 - 200

Product name	N_2 surface area (m^2/g)	V_2 (ml) = NaOH consumption (ml/g)
Hisil 255	170	16.9
KS 404	175	16.9
Ultrasil VN 3	175	20.7
Silica according to the invention (example 3)	184	15.7

The lower V_2 = NaOH consumption, the lower is the OH group density. The comparison shows that the silicas according to the invention may have a silanol group density of up to 40% lower than known precipitated silicas.

Example 6

Determination of average aggregate size by photon correlation spectroscopy

Parameters:

Ultrasound time: 15'

Suspending agent: Isopropanol/pentanol 10:1

Weight: 30 mg silica per 10 ml suspending agent

N_2 surface area (m^2/g): 100 - 150

Product name	N_2 surface area	Average

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	(m²/g)	aggregate size (nm)
Hisil 210	130	254
KS 300	125	197
Ultrasil VN 2	125	191
Silica according to the invention (example 2)	120	490

N_2 surface area (m2/g): 150 - 200

Product name	N_2 surface area (m²/g)	Average aggregate size (nm)
Hisil 255	170	152
KS 404	175	218
Ultrasil VN 3	175	167
Silica according to the invention (example 3)	184	381

The average aggregate size of the silicas according to the invention is distinctly greater than that of the known precipitated silicas.

Example 7

Silica according to Example 2 of the invention in comparison with Ultrasil VN 2 in a natural rubber formulation with Si 69

	1	2
First Latex Crepe	100	100
Ultrasil VN 2	50	–
Silica according to Example 2 of the invention	–	50
ZnO RS	4	4
Stearic acid	2	2
DEG	1	1
Si 69	3.2	3.2
CBS	1.6	1.6
TMTM	0.3	0.3
Sulfur	0.8	0.8
Mooney viscosity (MU)	77	69

Vulcanizate data: 150°C/$t_{95\%}$

	1	2
300% modulus (MPa)	8.1	9
Ball rebound (%)	56.8	58.6
DIN abrasion (mm3)	125	114
Goodrich flexometer (0.175", 108 N, RT, 18 h) T-center (°C)	81.2	70.8
MTS (DIN 53 513) tan δ /60°C		

In comparison with Ultrasil VN 2, which is comparable in surface area, the silica according to Example 2 of the invention leads to lower viscosities, higher modulus and resilience values, improved abrasion, lower heat build-up and lower loss angle tan at 60°C and thus lower rolling resistance.

Example 8

Silica according to the invention (Example 3) in comparison with Ultrasil VN 3 in a solution-SBR/BR tire tread formulation with Si 69

	1	2
VSL 1955 S 25	96	96
Buna CB 24	30	30
Ultrasil VN 3	80	–
Silica according to the invention (example 3)	–	80
ZnO RS	3	3
Stearic acid	2	2
Naftolen ZD	10	10
Vulkanox 4020	1.5	1.5
Protektor G 35	1	1
Si 69	6.4	6.4
CBS	1.5	1.5
DPG	2	2
ZBED	0.2	0.2
Sulfur	1.5	1.5
Mooney viscosity (MU)	72	68

Vulcanizate data: 150°C/t95%

	1	2
300% modulus (MPa)	8.9	9.3
Ball rebound (%)	52.6	54.7
MTS (DIN 53 513)		

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	1	2
tan δ 0°C	0.480	0.501
tan δ 0°C	0.152	0.144

In comparison with VN 3, the silica according to the invention has lower viscosity, higher modulus, higher elas-

5 ticity and, particularly importantly, higher wet skid resistance with low rolling resistance.

Example 9

Comparison of dispersion of silica according to the invention (Example 2) compared with VN 2 (identical N_2 surface

10 area of approx. 120 m^2/g) (using the Philipps methods, described in technical information brochure 102 A).

A piece of rubber approximately 20-30 μm in thickness (area approx. 5 x 5 mm) is cut using a Vibracut apparatus from FTB-Feinwerktechnik from a 6 mm sheet of vulcanizate of

15 the formulation according to example 8 filled with 80 parts of Ultrasil VN 2 or of the silica according to the invention from Example 2, related to 100 parts of rubber. This sample of rubber is transferred onto a glass slide and covered with a second glass slide. The sample prepared in this manner is

20 examined under a light microscope with a reflected light attachment and reproduced as a negative image with 55-times magnification. A positive image with the desired final magnification is prepared from this negative.

Dispersion is assessed according to the Philipps method

25 using 10 standard photographs as stated below:

Number	Dispersion
1 - 2	very poor
3 - 4	poor
5 - 6	adequate
7 - 8	good
9 - 10	very good

30

The dispersion of Ultrasil VN 2 is rated at 5 and is thus adequate, the dispersion of the silica according to the

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invention from Example 2 is rated at 9 and is thus very
good.

Example 10

Comparison of dispersion of silica according to the inven-

5 tion from Example 3 with Ultrasil VN 3 (identical N_2 surface
area of approx. 175 m^2/g)

Formulation, procedure and assessment are performed in a
similar manner to Example 9.

   The dispersion of Ultrasil VN 3 is rated at 2 and is

10 thus very poor, the dispersion of the silica according to
Example 3 of the invention is rated at 8 and is thus good.

Example 11

Determination of dispersion by roughness measurement using a
Federal Dispersion Analysis EM D-4000-W7 unit. Comparison

15 of Ultrasil VN 2 with the silica according to the invention
from Example 2.

   A piece of rubber (20 x 2 mm) is cut using a cutting
device also supplied by the above-stated equipment manufac-
turer from a 2 mm sheet of vulcanizate of the formulation

20 according to Example 8 filled with 80 parts of Ultrasil VN 2
or with the silica according to the invention from
Example 2, related to 100 parts of rubber, and is fixed into
a holder provided by the equipment manufacturer. The sur-
face of the vulcanizate is scanned using a diamond needle

25 and the surface roughness caused by dispersion so deter-
mined. This process allows dispersion to be quantified as
the device determines a value F^2H. F means the number of
peaks and H their average height. Thus, the lower the value
of this parameter, the better is the dispersion of the

30 filler in the vulcanizate sample.

For the above-stated fillers, the parameter F^2H gave the
following values:

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	Ultrasil VN 2	Silica according to the invention (example 2)
F^2H	82366	32556

The silica according to the invention thus has dis-

5 tinctly better dispersion. This method thus confirms the

results from Example 9.

Example 12

Comparison of dispersion of Ultrasil VN 3 with the silica

according to the invention from Example 3 using the rough-

10 ness measurement from Example 11.

The filling rate and procedure are here similar to those in

Example 11.

	Ultrasil VN 3	Silica according to the invention (example 3)
F^2H	55601	22602

15

The silica according to the invention has distinctly

better dispersion characteristics than VN 3. This method

confirms the results found in Example 10.

BRIEF DESCRIPTION OF FIGURES OF DRAWING

The drawings compare the essential physicochemical pa-

20 rameters of the precipitated silica according to the inven-

tion with those of known precipitated silica. The drawings

show:

Figure 1   the ratio of CTB to DBP

Figures 2 to 4 the ratio of CTAB to DBP

25

Figure 5   the ratio of CTAB to V2/V1

Figure 6   the ratio of CTAB to DBP/CTAB.

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